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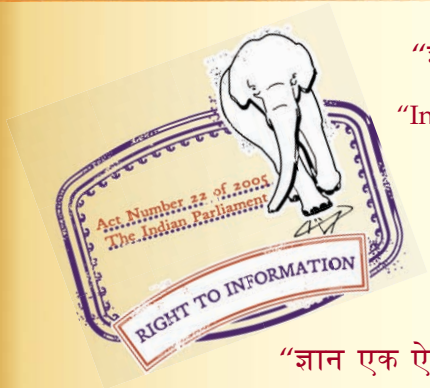
IS/ISO 9917-1 (2003): Dentistry - Water-based cement, Part 1: Powder/liquid acid-base cements [MHD 8: Dentistry]



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भाग 1 चूर्ण/द्रव अम्ल-क्षार सीमेंट

Indian Standard

DENTISTRY — WATER-BASED CEMENTS

PART 1 POWDER/LIQUID ACID-BASE CEMENTS

ICS 11.060.10

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Price Group 9

NATIONAL FOREWORD

This Indian Standard (Part 1) which is identical with ISO 9917-1 : 2003 'Dentistry — Water-based cements — Part 1: Powder/liquid acid-base cements' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendation of the Dentistry Sectional Committee and approval of the Medical Equipment and Hospital Planning Division Council.

Other part of this standard is as follows:

Part 2 Light-activated cements

The text of ISO Standard has been approved as suitable for publication as an Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.

b) Comma (,) has been used as a decimal marker while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

In this adopted standard, reference appears to certain International Standards for which Indian Standards also exist. The corresponding Indian Standards which are to be substituted in their respective places are listed below along with their degree of equivalence for the editions indicated:

International Standard	*Corresponding Indian Standard*	*Degree of Equivalence*
ISO 7491 : 1985 Dental materials — Determination of colour stability	IS 12181 : 1987 Determination of colour stability of dental polymeric materials	Technically Equivalent
ISO 10993-1:1992 Biological evaluation of medical devices — Part 1 : Evaluation and testing	IS 12572 (Part 1) : 1994 Biological evaluation of medical devices: Part 1 Guidance on selection of tests (*first revision*)	Identical
ISO 3696 : 1987 Water for analytical laboratory use — Specification and test methods	IS 1070 : 1992 Reagent Grade Water — Specification (*third revision*)	Technically Equivalent

The technical committee has reviewed the provisions of the following International Standards referred in this adopted standard, and has decided that they are acceptable for use in conjunction with this standard:

International Standard	*Title*
ISO 2590 : 1973	General method for the determination of arsenic — Silver diethyldithiocarbamate photometric method
ISO 3665 : 1996	Photography — Intra-oral dental radiographic film — Specification
ISO 7405 : 1997	Dentistry — Preclinical evaluation of biocompatibility of medical devices used in dentistry — Test methods for dental materials

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

DENTISTRY — WATER-BASED CEMENTS

PART 1 POWDER/LIQUID ACID-BASE CEMENTS

1 Scope

This part of ISO 9917 specifies requirements and test methods for powder/liquid acid-base dental cements intended for permanent cementation, lining and restoration. It is applicable to both hand-mixed cements and encapsulated cements for mechanical mixing. It specifies limits for each of the properties according to whether the cement is intended for use as a luting agent, a base or liner, or a restorative material.

Specific qualitative and quantitative requirements for freedom from biological hazard are not included in this part of ISO 9917, but it is recommended that, in assessing possible biological or toxicological hazards, reference should be made to ISO 7405, *Dentistry — Preclinical evaluation of biocompatibility of medical deviced used in dentistry — Test methods for dental materials*, and ISO 10993-1, *Biological evaluation of medical devices — Part 1: Evaluation and testing*.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 2590, *General method for the determination of arsenic — Silver diethyldithiocarbamate photometric method*

ISO 3665, *Photography — Intra-oral dental radiographic film — Specification*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 7491, *Dental materials — Determination of colour stability*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
mixing time
that part of the working time required in order to obtain a satisfactory mix of the components

3.2
working time
period of time, measured from the start of mixing, during which it is possible to manipulate a dental material without an adverse effect on its properties

3.3
net setting time
period of time, measured from the end of mixing, until the material has set according to the criteria and conditions specified in Annex A

NOTE For the purposes of this part of ISO 9917, in view of the wide variation in mixing times of cements, the net setting time is determined from the end of mixing.

4 Classification

4.1 Chemical type

For the purposes of this part of ISO 9917, dental cements shall be classified on the basis of their chemical composition, as follows:

a) zinc phosphate cement, see Clause B.1;

b) zinc polycarboxylate cement, see Clause B.2;

c) glass polyalkenoate cement, see Clause B.3.

Acid-base setting, water-based cements other than those listed above may fall within the scope of this part of ISO 9917. If the manufacturer wishes to claim conformity for a particular product, the manufacturer shall specify the type of material in accordance with 4.1 and 4.2 so that the correct performance limits are applied.

4.2 Application

For the purposes of this part of ISO 9917, materials shall be classified on the basis of their usage as follows:

a) luting cements;

b) bases or liners;

c) restorative cements.

5 Material

5.1 General

The cement shall consist of a powder and liquid which, when mixed in accordance with the manufacturer's instructions, shall conform with the requirements in this clause and in Clauses 8 to 15.

5.2 Components

5.2.1 Liquid

For non-encapsulated cements, visually inspect the liquid. It shall be free from deposits or filaments on the inside of its container. There shall be no visible signs of gelation.

5.2.2 Powder

For non-encapsulated cements, visually inspect the powder. It shall be free from extraneous material. If the powder is coloured, the pigment shall be uniformly dispersed throughout the powder.

Encapsulated cements shall not be examined in this way.

5.3 Unset cement

The cement shall be mixed in accordance with Clause 6, and then visually inspected. It shall be homogeneous and of a smooth consistency.

6 Preparation of test specimens

6.1 Ambient conditions

All specimens shall be prepared at a temperature of (23 ± 1) °C and a relative humidity of (50 ± 10) %.

6.2 Method of mixing

The cement shall be prepared in accordance with the manufacturer's instructions. Sufficient cement shall be mixed to ensure that the preparation of each specimen is completed from one mix. A fresh mix shall be prepared for each specimen.

NOTE For encapsulated materials, more than one capsule simultaneously mixed may be required for certain specimens.

7 Sampling

7.1 Hand-mixed cements

The sample, drawn from one batch, shall provide sufficient material to complete all the prescribed tests and any necessary repeats.

NOTE Approximately 50 g of powder is necessary to complete the tests.

7.2 Encapsulated cements

The sample shall comprise a retail package of a sufficient number of capsules from one batch to complete all the tests.

8 Net setting time

When determined in accordance with Annex A, the net setting time of the cement shall conform to relevant requirements specified in Table 1.

9 Film thickness (luting cements only)

When determined in accordance with Annex C, the film thickness shall conform to relevant requirements specified in Table 1.

10 Compressive strength

When determined in accordance with Annex D, the compressive strength of the cement shall conform to the relevant requirements specified in Table 1.

11 Acid erosion

When determined in accordance with Annex E, the acid erosion of the cement shall conform to the relevant requirements specified in Table 1.

12 Optical properties (polyalkenoate restorative cements only)

When prepared, stored and tested in accordance with Annex F, cements shall meet the following requirements:

a) The opacity of the set restorative cement shall be within the limits specified in Table 1, unless the restorative cement is designated as opaque by the manufacturer (see Clause 15).

b) The colour of the set cement shall match the manufacturer's shade guide. If a shade guide is not supplied by the manufacturer, then the manufacturer shall nominate a commercially available shade guide [see 15.3.1 c)].

13 Acid-soluble arsenic and lead contents

13.1 Acid-soluble arsenic content

When determined in accordance with Annex G, the acid-soluble arsenic content shall not exceed the relevant limits specified in Table 1.

13.2 Acid-soluble lead content

When determined in accordance with Annex G, the acid-soluble lead content shall not exceed the relevant limits specified in Table 1.

14 Radiopacity

When the manufacturer describes the material as being radiopaque (see 15.2), the radiopacity shall be at least equivalent to that of the same thickness of aluminium when determined in accordance with Annex H. When the manufacturer claims a greater radiopacity, the measured value shall not be less than the value claimed when determined in accordance with Annex H.

15 Packaging, marking and information to be supplied by manufacturer

NOTE Additional information may be included at the discretion of the manufacturer or as required by legislation.

15.1 Packaging

The materials shall be supplied in containers or capsules (for the purposes of this part of ISO 9917, the container or capsule shall be considered to be the immediate wrapping of the material) that afford adequate protection and have no adverse effect on the quality of the contents.

An outer pack may also be used to present the containers or capsules as a single unit.

15.2 Marking

Each outer container shall be clearly marked with the following details:

a) the name and/or trademark of the manufacturer and the trade name, type and application of the cement;

b) the shade of the powder according to the manufacturer's nominated shade guide;

c) the minimum net mass, in grams, of the powder or the minimum net volume, in millilitres, of the liquid, as appropriate;

d) the manufacturer's batch or lot number;

e) on the outermost packaging, the recommended conditions of storage, and the "expiry date" for the material under those conditions of storage;

f) if the cement is designated opaque, the outer packaging shall be clearly marked with this information;

g) if the cement is designated as radiopaque, the outer packaging shall be clearly marked with this information;

h) each outer container of encapsulated cements shall be marked with the number of capsules in the container, and the net mass in each capsule.

15.3 Manufacturer's instructions

15.3.1 General

Instructions shall accompany each package of the material and shall include the following:

a) the trade name of the product;

b) the manufacturer's name and contact details;

c) a shade guide or, when no shade guide is provided, details of a commercially available shade guide for use with the material.

In addition, at least the information in 15.3.2 or 15.3.3, as appropriate, shall be given. For those materials not mentioned specifically in 4.1, the manufacturer shall indicate the type of material (see 4.1 and 4.2) against whose performance requirements the material shall be tested.

15.3.2 Hand-mixed cements

For hand-mixed cements, the following information shall be supplied:

a) the temperature range for preparation;

b) the recommended powder/liquid ratio, expressed as a mass/mass ratio for the recommended temperature range, and an indication of how the user may achieve this ratio [also, for test purposes, the powder/liquid ratio on a mass/mass basis to a precision of 0,01 g at a temperature of (23 ± 1) °C and a relative humidity of (50 ± 10) %];

c) the type of mixing slab and spatula and their condition;

d) the rate of incorporation of the powder into the liquid;

e) the mixing time (see 3.1);

f) the working time (see 3.2);

g) the net setting time (see 3.3);

h) if appropriate, a statement recommending that a liner should be placed between the cement and the dentine;

i) if appropriate, the minimum time at which finishing may be commenced and the recommended method of finishing;

j) if appropriate, a statement indicating that the surface of the cement must be coated with a protective coating and guidance on the type of coating to be used.

15.3.3 Encapsulated cements

For encapsulated cements, the following instructions shall be supplied:

a) the method of bringing about physical contact between the powder and liquid;

b) the type of mechanical mixing device and the mixing time to be employed;

c) the net setting time (see 3.3);

d) the working time (see 3.2);

e) if appropriate, a statement recommending that a liner should be placed between the cement and the dentine;

f) if appropriate, the minimum time at which finishing may be commenced and the recommended method of finishing;

g) the minimum deliverable volume, in millilitres, of mixed cement in one capsule;

h) if appropriate, a statement indicating that the surface of the cement must be coated with a protective coating and guidance on the type of coating to be used.

Table 1 — Requirements for dental cements

Chemical type	Application	Film thickness	Net setting time		Compressive strength	Acid erosion	Opacity $C_{0,70}$		Acid-soluble As content	Acid-soluble Pb content
		µm	minutes		MPa	mm			mg/kg	mg/kg
		max.	min.	max.	min.	max.	min.	max.	max.	max.
Zinc phosphate	Luting	25	2,5	8	50	0,30			2	100
Zinc polycarboxylate	Luting	25	2,5	8	50	0,40			2	100
Glass polyalkenoate	Luting	25	1,5	8	50	0,17				100
Zinc phosphate	Base/liner		2	6	50	0,30			2	100
Zinc polycarboxylate	Base/liner		2	6	50	0,40			2	100
Glass polyalkenoate	Base/liner		1,5	6	50	0,17				100
Glass polyalkenoate	Restorative		1,5	6	100	0,17	0,35	0,90		100

Annex A
(normative)

Determination of net setting time

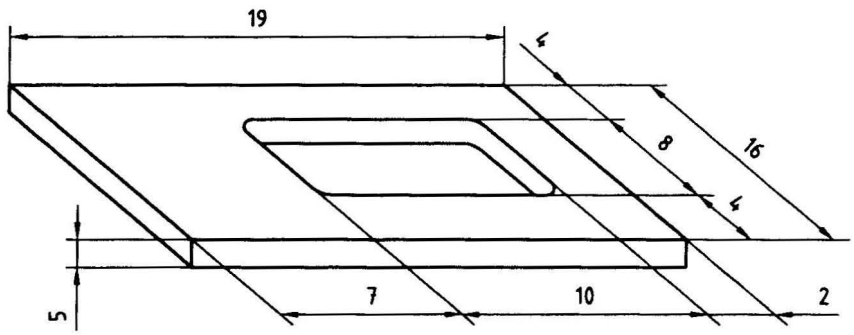
A.1 Apparatus

A.1.1 Cabinet, capable of being maintained at a temperature of (37 ± 1) °C and a relative humidity of at least 90 %.

A.1.2 Indentor, of mass (400 ± 5) g, with a needle having a flat end of diameter (1,0 ± 0,1) mm which is plane and perpendicular to the long axis of the needle.

A.1.3 Metal mould, similar to that shown in Figure A.1.

Dimensions in millimetres
Tolerance on dimensions: ± 0,15



NOTE Internal corners may be square or rounded.

Figure A.1 — Mould for preparation of specimens for determination of net setting time

A.1.4 Metal block, of minimum dimensions 8 mm × 75 mm × 100 mm, positioned within the cabinet and maintained at (37 ± 1) °C.

A.1.5 Aluminium foil.

A.1.6 Timer, accurate to 1 s.

A.2 Procedure

Place the mould (A.1.3), conditioned to (23 ± 1) °C, on the aluminium foil (A.1.5) and fill to a level surface with mixed cement.

60 s after the end of mixing, place the assembly, comprising mould, foil and cement specimen, on the block (A.1.4), in the cabinet (A.1.1). Ensure good contact between the mould, foil and block.

90 s after end of mixing, carefully lower the indentor (A.1.2) vertically onto the surface of the cement and allow it to remain there for 5 s. Carry out a trial run to determine the approximate setting time, repeating the indentations at 30 s intervals until the needle fails to make a complete circular indentation in the cement, when viewed using × 2 magnification. Clean the needle, if necessary, between indentations. Repeat the process, starting the indentation at 30 s before the approximate setting time thus determined, making indentations at 10 s intervals.

Record the net setting time as the time elapsed between the end of mixing and the time when the needle fails to make a complete circular indentation in the cement. Repeat the test two more times.

A.3 Expression of results

Record the results of the three tests. Each result shall fall within the range specified in Table 1 for the material to meet the requirement.

Annex B
(informative)

Chemical compositions and applications of dental cements

B.1 Zinc phosphate cements

Zinc phosphate cements are based on the reaction between an oxide powder (the principal constituent of which is zinc oxide) and an aqueous solution of phosphoric acid which may contain metal ions. They can be used to cement dental appliances to hard oral structures or to other appliances. They can also be used as a base for restorative materials and as a temporary restorative material.

B.2 Zinc polycarboxylate cements

Zinc polycarboxylate cements are based on the reaction between zinc oxide and aqueous solutions of polyacrylic acid or similar polycarboxylic compounds, or zinc oxide/polycarboxylic acid powders which are mixed with water. They can be used to cement dental appliances to hard oral structures or to other appliances. They can also be used as a base for restorative materials and as a temporary restorative material.

B.3 Glass polyalkenoate cements

Glass polyalkenoate (glass ionomer) cements are based on the reaction between an aluminosilicate glass powder and an aqueous solution of a polyalkenoic acid, or between an aluminosilicate glass/polyalkenoic acid powder blend and water or an aqueous solution of tartaric acid. These cements are used for the aesthetic restorations of teeth or as bases or liners for other restorations. They can also be used to cement dental appliances to hard oral structures or to other appliances. This type also includes glass polyalkenoate cements in which the glass and a metal have been fused (cermets) or mixed together and which are intended for the restoration of teeth.

Annex C
(normative)

Determination of film thickness (luting cements only)

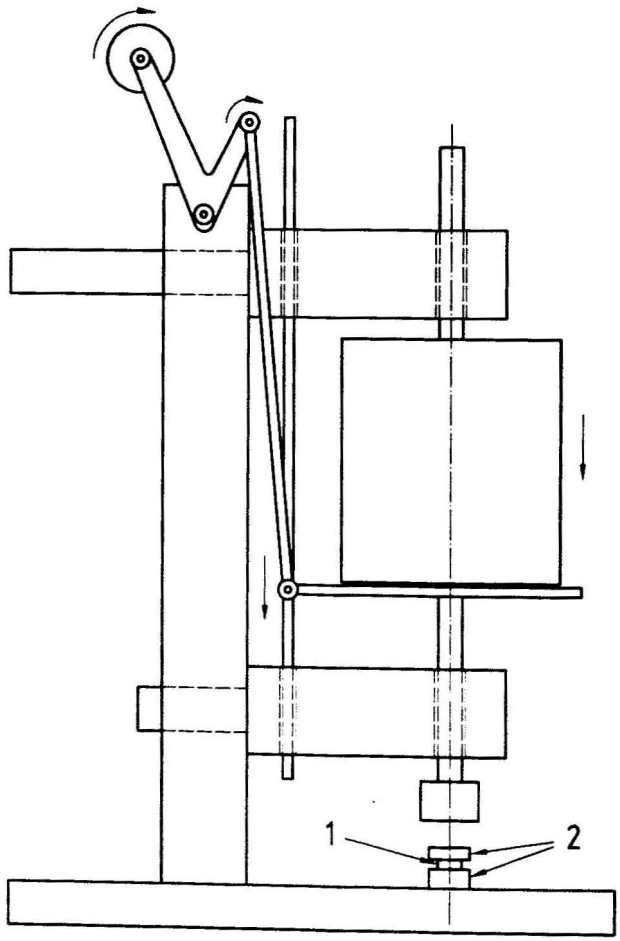
C.1 Apparatus

C.1.1 Two optically flat, square or circular glass plates, having a contact surface area of (200 ± 25) mm^2. Each plate shall be of a uniform thickness of not less than 5 mm.

C.1.2 Loading device, of the type illustrated in Figure C.1, or an equivalent means, whereby a force of (150 ± 2) N may be generated vertically to the specimen via the upper glass plate.

The anvil, which is attached to the bottom of the rod carrying the load, shall be horizontal and parallel to the base. The device shall be such that the force is applied smoothly and in such a manner that no rotation occurs.

C.1.3 Screw micrometer or **equivalent measuring instrument**, having graduations of 2 µm or smaller.



Key

1 specimen
2 glass discs

Figure C.1 — Loading device for use in film thickness test

C.2 Procedure

Measure and record to an accuracy of 1 µm the combined thickness of the two optically flat glass plates (C.1.1) stacked in contact and designate this measurement reading A. Remove the upper plate, place (0,10 ± 0,05) ml of the mixed cement in the centre of the lower plate and place this centrally below the loading device (C.1.2) on the lower platen. Replace the second glass plate centrally on the cement in the same orientation as in the original measurement.

Ten seconds before the end of the manufacturer's stated working time, carefully generate a force of (150 ± 2) N vertically and centrally to the specimen via the top plate. Ensure that the cement has completely filled the space between the glass plates. When at least 10 min have elapsed after the application of the force, remove the plates from the loading device and measure the combined thickness of the two glass plates and the cement film. Designate this measurement reading B.

Record the difference in thickness of the plates with and without the cement film (reading B – reading A) as the thickness of the film. Repeat the test four times.

C.3 Treatment of results

At least four of the five results shall be below 25 µm for the material to pass the requirement specified in Table 1. If only two or less results are below 25 µm, then the material shall fail the requirement.

If three results are below 25 µm, a further five specimens shall be tested. To comply with the requirement specified in Table 1, all the specimens in the second series shall be below 25 µm.

Annex D
(normative)

Determination of compressive strength

D.1 Apparatus

D.1.1 Cabinet, maintained at a temperature of (37 ± 1) °C and a relative humidity of at least 30 %.

D.1.2 Split mould and plates, as shown in Figure D.1. The mould shall have internal dimensions (6,0 ± 0,1) mm high and (4,0 ± 0,1) mm diameter. The mould and plates shall be made of stainless steel or a material which will not be affected by the cement. When testing polyacid-based cements, face the plates with acetate sheets to prevent adhesion.

D.1.3 Screw clamp, as shown in Figure D.1.

D.1.4 Screw micrometer or **equivalent measuring instrument**, having graduations of 2 µm or smaller.

D.1.5 Mechanical tester, which is capable of being operated at a cross-head speed of (0,75 ± 0,30) mm/min or at a loading rate of (50 ± 16) N/min.

Dimensions in millimetres

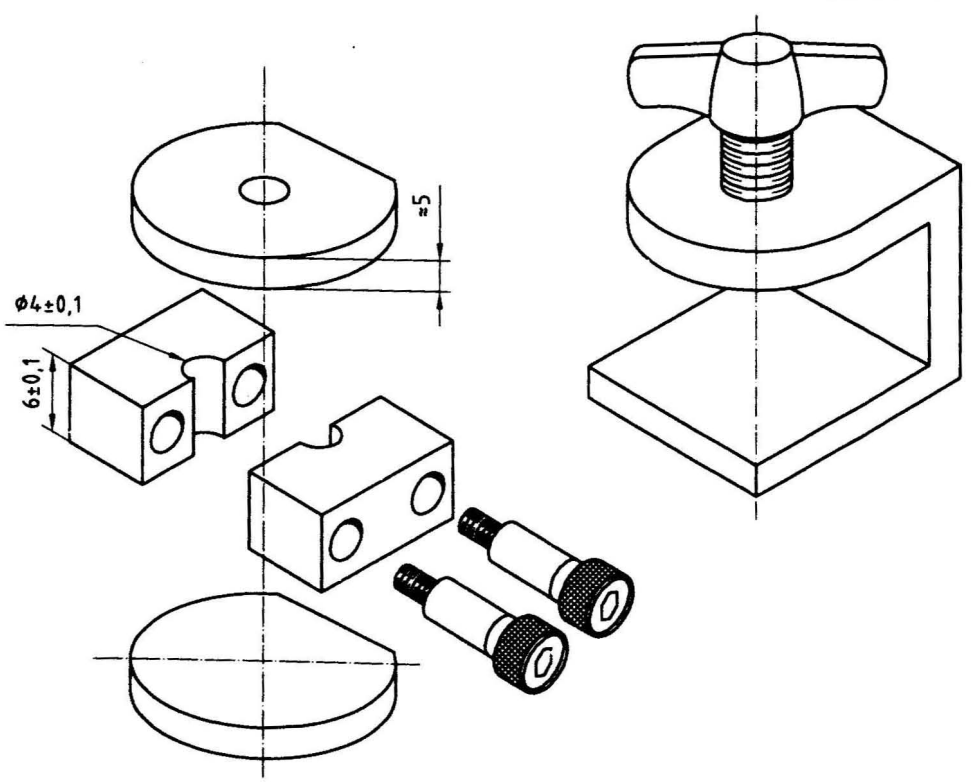


Figure D.1 — Mould and clamp for preparation of specimens for compressive strength test

D.2 Preparation of test specimens

Condition the split mould and plates (D.1.2) and screw clamp (D.1.3) at (23 ± 1) °C. Within 60 s after the end of mixing, pack the cement, prepared in accordance with the manufacturer's instructions, to a slight excess in the split mould.

In order to consolidate the cement and avoid trapping air, convey the largest convenient portions of mixed cement to the mould and apply to one side with a suitable instrument. Fill the mould to excess in this manner and then place on the bottom plate with some pressure.

Remove any bulk extruded cement, place the top metal plate in position on the mould and squeeze together. Put the mould and plates in the screw clamp and tighten. Not later than 120 s after the end of mixing, transfer the whole assembly to the cabinet (D.1.1).

One hour after the end of mixing, remove the plates and grind the ends of the specimen flat and at right angles to its long axis. An acceptable method for doing this is to use wet 400 grade silicon carbide paper, but in any event the abrasive shall be no coarser.

Remove the specimen from the mould immediately after surfacing and check visually without magnification for air-voids or chipped edges. Discard any such defective specimens.

NOTE To facilitate the removal of the hardened cement specimen, the internal surface of the mould may be evenly coated, prior to filling, with a 3 % solution of micro-crystalline or paraffin wax in petroleum ether. Excess ether is allowed to evaporate before using the mould. Alternatively, a thin film of silicone grease or PTFE dry-film lubricant may be used.

Prepare five such specimens and, immediately after the preparation of each, immerse it in water, grade 3 as defined in ISO 3696, at (37 ± 1) °C for (23 ± 0,5) h.

Calculate the diameter of each specimen by taking the mean of two measurements at right angles to each other made to an accuracy of ± 0,01 mm using e.g. a screw micrometer (see D.1.4).

D.3 Procedure

Twenty-four hours after the end of mixing, place each specimen with the flat ends between the plates of the mechanical tester (D.1.5) and apply a compressive load along the long axis of the specimen. Apply a sheet of damp filter paper (e.g. Whatman No. 1) to both top and bottom platens of the test machine in the area which will contact the specimens. Use a fresh piece of paper for each test.

Record the maximum force applied when the specimen fractures and calculate the compressive strength, C, in megapascals using the equation:

$$C = \frac{4p}{\pi d^2}$$

where

p is the maximum force applied, in newtons;

d is the measured diameter of the specimen, in millimetres.

D.4 Treatment of results

If at least four of the five results are above the minimum strength specified in Table 1, the material shall pass the test. If three or more of the five results obtained are below the minimum strength specified in Table 1, the material shall fail the test.

If only three specimens satisfy the minimum strength requirement in Table 1, prepare and test a further five specimens. To pass the test, at least eight of the total of ten results shall be above the minimum strength value specified in Table 1.

Annex E
(normative)

Determination of acid erosion

E.1 Apparatus

E.1.1 Cabinet, capable of being maintained at a temperature of (37 ± 1) °C and a relative humidity of at least 90 %.

E.1.2 Specimen-holders, comprising squares or discs of cast poly(methyl methacrylate) (PMMA) (30 mm × 30 mm × 5 mm squares or 5 mm × 30 mm diameter discs), with a hole (5,0 ± 0,5) mm in diameter × (2,0 ± 0,5) mm deep bored in the centre.

NOTE The size and shape of the specimen-holder can be slightly changed (e.g. corners of squares rounded) if this helps it to fit comfortably into the container.

E.1.3 PMMA plate.

NOTE The size of the PMMA plate is not important but is approximately the same as that of the specimen holder.

E.1.4 Separating sheet, such as a polyester strip, for application to the specimen-holder to facilitate removal of the PMMA plate after the cement has set.

E.1.5 Individual clamps, clips or **equivalent devices**, designed to hold the specimen mould together during setting of the cement.

E.1.6 Balance, with an accuracy of 0,1 mg.

E.1.7 Volume-measuring device such as a pipette, suitable for 30 ml aliquots, with an accuracy of 0,1 ml.

E.1.8 Container, capable of carrying 30 ml of test solution in which the specimen-holder can be completely immersed horizontally and in which the distance between the surface of the eroding solution and the upper surface of the immersed specimen-holder is (10 ± 3) mm. The container shall have a lid or screw cap to prevent evaporation or contamination.

E.1.9 Cabinet, maintained at a temperature (37 ± 1) °C.

NOTE The cabinet (E.1.1) may be used but 90 % humidity is not required here.

E.1.10 Abrasive paper: 1 200 grit wet or dry abrasive paper.

E.1.11 Spring-loaded, spherically tipped, dial gauge, having a scale graduated in units of 0,01 mm or less (to enable 0,005 mm to be measured). The spring force of the gauge shall be in the range 50 g to 100 g and the diameter of the tip shall be (3,5 ± 0,5) mm.

NOTE It is acceptable to use an equivalent loading device with a "deadweight". A device with an electronic reading/display facility is also acceptable providing the loading conditions are as specified.

E.2 Preparation of eroding solution

Dissolve 8,26 g of lactic acid (general purpose reagent grade or purer) and 0,92 g of sodium lactate (general purpose reagent grade or purer), both calculated as pure, dry substance, in water (grade 3 as defined in ISO 3696) and adjust to 1 l with grade 3 water, at least 18 h before use (this allows hydrolysis of lactone to take place). Immediately prior to use, check that the pH of this solution is 2,74 ± 0,02 and adjust if necessary with 1 mol/l sodium lactate solution or 1 mol/l lactic acid solution. This reagent shall always be prepared freshly for each set of specimens being tested.

NOTE This solution is a 0,1 mol/l lactic acid/sodium lactate buffer solution. The pH is 2,74.

E.3 Preparation of test specimens

Condition the specimen-holders (E.1.2), PMMA plate (E.1.3), separating sheet (E.1.4) and clamp (E.1.5) at (23 ± 2) °C. Proportion and mix the test material in accordance with the manufacturer's instructions at (23 ± 2) °C. For hand-mixed cements, weigh powder and liquid at the recommended powder/liquid ratio by using the balance (E.1.6). Place the components on the mixing pad/slab recommended by the manufacturer. Mix using the technique recommended by the manufacturer. For encapsulated cements, mix by using the recommended mixing machine for the recommended time. Within 60 s after the end of mixing of the test material, fill the hole in the specimen-holder with the cement following the manufacturer's instructions for handling.

In order to avoid trapping air, use the largest convenient portions of mixed cement to fill one side of the hole in the specimen-holder then fill the remainder of the specimen holder, continuing to avoid air entrapment. Fill the specimen-holder to a slight excess in this manner.

Cover the cement with the PMMA plate, faced with the separating sheet, press firmly together and apply the screw clamp.

At 180 s after the end of mixing, transfer the whole assembly to the cabinet (E.1.1) maintained at (37 ± 1) °C and a relative humidity of at least 90 %.

After 24 h, remove the plate and the separating sheet from the clamp and lap the cement in the specimen holder until flat using the abrasive paper (E.1.10) with continuous water irrigation.

Prepare five specimens. Inspect all specimens and reject any with obvious flaws or air inclusions.

E.4 Measurement and calculation of initial depth at centre of cement, D_0

For each specimen, measure the initial depth at the centre of the specimen using the edge of the specimen-holder as a fixed reference plane.

NOTE 1 This procedure is necessary to establish the initial cement height and to confirm that the height of the cement is approximately the same as the height of the specimen-holder.

If the original cement height is not within 5 µm of that of the specimen holder, carry out further lapping until the levels of the cement and holder are within 5 µm.

NOTE 2 Typically, one unit of the dial gauge is 10 µm and 5 µm can be estimated as a half of one unit.

Record the height at four points on the specimen holder and near the centre of the material as a baseline depth using the dial gauge (E.1.11). The four points on the specimen-holder shall be spaced evenly at 90° intervals at 0,5 mm to 1,0 mm from the specimen.

Calculate the average of the four heights of the specimen holder. Subtract the height at the centre of the specimen from this value to obtain D_0.

E.5 Immersion

Immerse each specimen with its specimen-holder horizontally in an individual container (E.1.8) containing 30 ml of the eroding solution. Place the specimen holder "face upwards" so that the whole of the cement surface is bathed in solution with a (10 ± 3) mm head of solution above the surface of the test specimen.

Annex F
(normative)

Determination of optical properties (polyalkenoate restorative cements only)

F.1 Opacity

F.1.1 Apparatus

F.1.1.1 **Cabinet**, maintained at a temperature of (37 ± 1) °C and a relative humidity of at least 30 %

F.1.1.2 **Opal glass standards**, with $C_{0,70}$ values of 0,35 and 0,55 and 0,90.

NOTE The contrast ratio $C_{0,70}$ is the ratio between the light reflected by the specimen on a black background and the light reflected by the specimen on a white background which has a diffuse reflectance of 70 %.

F.1.1.3 **Sheets of white waterproof material made of polyethylene or cellulose acetate**, measuring approximately 110 mm × 40 mm, marked, along their entire length, with black stripes 2 mm wide and 3 mm apart.

F.1.1.4 **Mould**, consisting of a split brass or stainless-steel ring contained in a former and cover plates as shown in Figure F.1. The height of the ring shall be (1,00 ± 0,03) mm and the internal diameter (10,0 ± 0,3) mm.

F.1.1.5 **Individual clamps, clips** or **equivalent devices**, designed to hold the specimen mould together during setting of the cement.

F.1.1.6 **Screw micrometer**, or **equivalent device**, accurate to 0,01 mm.

F.1.1.7 **Timer**, accurate to 1 s.

F.1.2 Preparation of test specimen

Place the mould on the sheet (F.1.1.3) backed by a flat metal cover plate. Fill the split ring mould (F.1.1.4) with a cement prepared in accordance with the manufacturer's instructions. Cover with a second plate faced with a sheet; press firmly together and hold together with a clamp (F.1.1.5).

120 s after the end of mixing, place the mould plates and the screw clamp in the humidity cabinet (F.1.1.1).

After 1 h, remove the plates and sheets from the clamp and carefully separate the specimen from the ring. Measure the thickness of the specimen using the micrometer (F.1.1.6) and use only specimens (1,0 ± 0,1) mm thick. Store the specimen for seven days at (37 ± 1) °C in water of grade 3 as defined in ISO 3696.

F.1.3 Procedure

Compare the opacity of the specimen with the two appropriate opal glass standards (F.1.1.2) by placing the specimen and the standards on the black and white striped background. Cover the specimen, the opal glass standards and the sheets with a thin film of distilled water while making the comparison.

If the opacity of the cement specimen is between those of the two standards or equal to either of them, it passes the test.

Alternatively, use a photometric instrument to make this comparison, provided that it can be demonstrated that the instrument has an accuracy of within ± 0,02 $C_{0,70}$. In this procedure, place the specimen on an illuminated white background (reflectance 70 %) and measure the reflectance, $R_{0,70}$. Then transfer it to a black background and illuminate it with the same source and measure the reflectance R_B. Calculate the opacity from the equation:

$$C_{0,70} = R_B / R_{0,70}$$

F.2 Colour

F.2.1 Apparatus

F.2.1.1 Sheet of white bond paper, giving a diffuse white background with a diffuse reflectance of approximately 90 %.

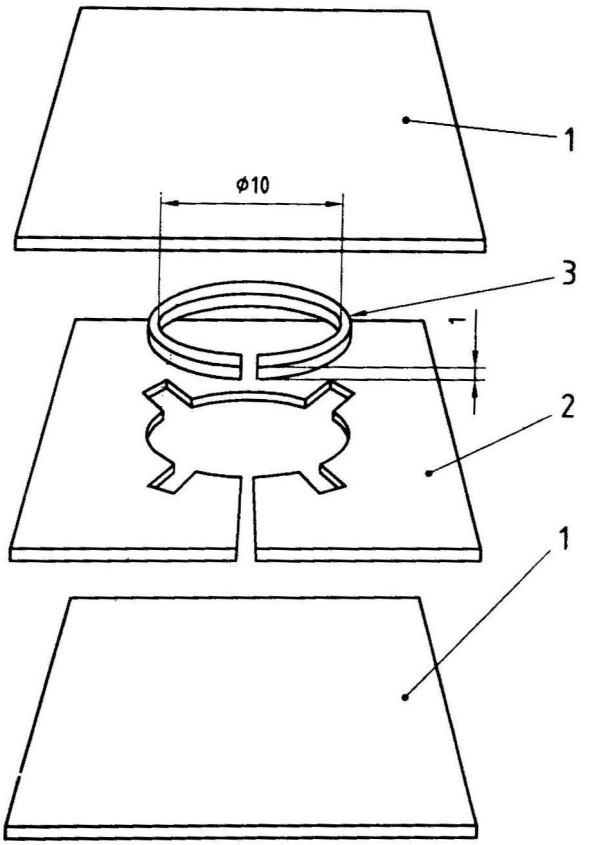
F.2.2 Preparation of test specimen

Prepare the test specimen as described in F.1.2 and store it at (37 ± 1) °C for seven days in water of grade 3 as defined in ISO 3696.

F.2.3 Procedure

Remove the specimen from the water. Remove the excess water from the cement surface with filter-paper and place the specimen on the diffuse white background. Compare the colour of the specimen with the manufacturer's shade guide or nominated shade guide using the procedure for colour comparison specified in ISO 7491. The colour of the specimen shall correspond with the manufacturer's description.

Dimensions in millimetres



Key

1 cover plate
2 retaining plate or former
3 split ring

Figure F.1 — Mould for preparation of specimens for opacity and colour test

Annex G
(normative)

Determination of acid-soluble arsenic and lead contents

G.1 Reagents

The reagents shall be of recognized analytical grade and "low in lead".

G.1.1 Hydrochloric acid, low in lead, concentrated (ρ = 1,18 g/ml).

G.1.2 Hydrochloric acid, dilute. Prepare by adding 20 ml of concentrated hydrochloric acid (G.1.1) to 80 ml of water (G.1.3).

G.1.3 Water, of grade 2 as defined in ISO 3696.

G.2 Preparation of sample

Mix sufficient powder and liquid, at the recommended powder/liquid ratio, to give 3 g of cement. Place the mixed cement in a clean plastic bag and seal the bag. Flatten the cement in the bag using finger pressure to produce a very thin disc. Place the disc in an oven at 37 °C for 24 h. After 24 h, crush the disc to a fine powder in an agate pestle and mortar. Accurately weigh about 2 g of the powdered cement and transfer to a 150 ml conical flask. Add 50 ml of the dilute hydrochloric acid (G.1.2). Stopper the flask, shake it and allow it to stand for 16 h.

Pour the solution into a centrifuge tube and centrifuge at [illegible] Decant the clear supernatant liquid into a suitable container and retain.

G.3 Procedure for arsenic determination

[illegible]

G.4 Procedure for lead determination

[illegible]

Annex H
(normative)

Determination of radiopacity

H.1 Apparatus

H.1.1 Single-phase dental X-ray unit, with a total filtration of 1,5 mm of aluminium and capable of operation at (65 ± 5) kV, with suitable accessories.

H.1.2 Dental X-ray occlusal film, of speed group D (as specified in ISO 3665), **freshly prepared developing solution** and **fixer**.

H.1.3 Aluminium step wedge, having a thickness range from 0,5 mm to 5,0 mm in equally spaced steps of 0,5 mm. The wedge shall be free-standing.

The aluminium alloy used to construct the wedge shall have an aluminium content of at least 98 mass %, a copper content of less than 0,1 mass % and an iron content of less than 1,0 mass %.

H.1.4 Photographic densitometer, capable of measuring in the optical density range 0,5 to 2,5.

H.1.5 Mould, for the preparation of specimen discs (15 ± 1) mm in diameter and (1,0 ± 0,1) mm thick (e.g. a metal washer).

H.1.6 Film, (50 ± 30) µm thick, made of e.g. polyester.

H.1.7 Glass or metal slides/plates (see D.1.2).

H.1.8 Cabinet, capable of being maintained at a temperature of (37 ± 1) °C and a relative humidity of at least 90 %.

H.1.9 Screw micrometer, or **equivalent device**, accurate to 0,01 mm.

H.1.10 Individual clamps, clips or **equivalent devices**, designed to hold the specimen mould together during setting of the cement.

H.1.11 Abrasive paper: 1 200 grit wet or dry abrasive paper.

H.2 Preparation of test specimens

Place a sheet of film (H.1.6) on a plate (H.1.7). Place the mould (H.1.5) on the film. Slightly overfill the mould (H.1.5) with cement. Place a piece of film (H.1.6) on the material in the mould and cover this with a second plate (H.1.7), thus expressing excess material. Secure the assembly in a clamp (H.1.10) to ensure that a specimen of the correct thickness is produced. Place the assembly in the cabinet (H.1.8) and allow to set for 30 min.

Remove the specimen from the mould and measure the thickness of the disc near its centre with the micrometer (H.1.9). Use only specimens whose thickness falls in the range (1,0 ± 0,1) mm. If the specimens are oversized, they may be sequentially ground using abrasive paper (H.1.11) until they fall within the specified range of thickness. Store in water of grade 3 as defined in ISO 3696 at (23 ± 1) °C for no more than 7 days before testing. In order to avoid dehydration of the test specimen, make the determination of radiopacity within 30 min of removing the specimen from the water.

H.3 Procedure

Position the X-ray film (H.1.2) on a sheet of lead not less than 2 mm thick. Place the specimen and the aluminium step wedge (H.1.3) in the centre of the film.

Irradiate the specimen, aluminium step wedge and film with X-rays at (65 ± 5) kV at a cathode-target film distance of 400 mm for a time such that, after processing, the region of film beside the specimen and aluminium has a photographic density of between 1,5 and 2.

NOTE 1 Exposures of between 0,3 s and 0,4 s at 10 mA are typical.

After developing and fixing the film, compare the density of the image of the specimen with that of the aluminium wedge using the densitometer (H.1.4)

NOTE 2 Improvement in the accuracy of this determination may be obtained by plotting a graph of optical density against thickness of aluminium for each exposure of the step wedge.

H.4 Interpretation of results

NOTE In interpreting the results of the radiopacity evaluation, it should be remembered that greater radiopacity produces a less dense (more transparent) image on the processed film.

The equivalent thickness of aluminium shall be estimated from the film (take the nearest higher value of thickness of the aluminium wedge: e.g. if the specimen produces a film density equivalent to between 3,5 mm and 4,0 mm of aluminium, the value of 4,0 mm is used).

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BUREAU OF INDIAN STANDARDS

Headquarters:

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Regional Offices: Telephones

Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002 — 2323 7617, 2323 3841

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